

**Orthophosphate or Total Phosphorus****SM 4500-P E – 1999 (2011)**

*ADDITIONAL QC REQUIREMENTS FOR THIS METHOD: Certified or Accredited laboratories using this method are assessed to applicable requirements of SM 1020 and SM 4020.*

Facility Name: \_\_\_\_\_ LAB ID: \_\_\_\_\_  
 Assessor Name: \_\_\_\_\_ Analyst Name: \_\_\_\_\_ Inspection Date: \_\_\_\_\_

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/Revision/Date: _____ Analyst: _____					
Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____					
1) If analyzing for dissolved phosphorus, are samples filtered immediately after collection and preserved by freezing at or below -10°C?	4500-P A.5				
2) If analyzing for orthophosphate, are samples filtered within 15 minutes of collection, preserved by storing at ≤6°C, and analyzed within 48 hours of collection?	40 CFR 136.3 Table 1I				
3) If analyzing for total phosphorus, are samples preserved to pH <2 with sulfuric acid, preserved by storing at ≤6°C, and analyzed within 28 days of collection?	40 CFR 136.3 Table 1I				
4) Is glassware acid-washed with HCl and rinsed with distilled water? <i>NOTE: If glassware is designated only for phosphate analysis, acid-washing may be done occasionally.</i>	4500-P E.2.b, C.2.b				
5) If analyzing for total phosphorus, are samples digested using persulfate digestion (SM 4500-P B.5)?	40 CFR 136.3 Table 1B				
<b>Persulfate digestion:</b>					
6) Is one of the following used? - Hotplate ("30 x 50 cm heating surface is adequate") - Autoclave or pressure cooker capable of 98-137 kPa	4500-P B.5.a				
7) Does the laboratory add to "50 mL or a suitable portion of thoroughly mixed sample" the following reagents? - 0.05 mL (1 drop) phenolphthalein indicator solution - If red color, H <sub>2</sub> SO <sub>4</sub> solution dropwise until color discharged - Then, 1 mL H <sub>2</sub> SO <sub>4</sub> solution - 0.4 g solid (NH <sub>4</sub> ) <sub>2</sub> S <sub>2</sub> O <sub>8</sub> OR 0.5 g solid K <sub>2</sub> S <sub>2</sub> O <sub>8</sub>	4500-P B.5.c				
8) Are treated samples either (a) boiled gently on a preheated hotplate for 30-40 minutes or until a final volume of 10mL is reached, cooled, and diluted to 30 mL with distilled water or (b) heated for 30 minutes in an autoclave or pressure cooker at 98-137 kPa and cooled?	4500-P B.5.c				
Notes/Comments:          					

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9) After digestion, is 0.05 mL (1 drop) phenolphthalein indicator solution added and are samples neutralized to a faint pink color with NaOH?	4500-P B.5.c				
10) After neutralizing, are samples diluted to 100 mL with distilled water?	4500-P B.5.c				
<b>Final determination of orthophosphate or total phosphorus:</b>					
11) Prior to analysis for <b>orthophosphate</b> , is 0.05 mL (1 drop) phenolphthalein indicator solution added to 50 mL sample, and if a red color develops, 5N H <sub>2</sub> SO <sub>4</sub> added dropwise until the color discharged?	4500-P E.4.a				
12) Is the combined reagent made fresh using the following proportions for 100 mL of the combined reagent? 50 mL 5N H <sub>2</sub> SO <sub>4</sub> , 5 mL potassium antimonyl tartrate solution, 15 mL ammonium molybdate solution, and 30 mL ascorbic acid solution in the order listed?	4500-P E.3.e				
13) Is 8 mL of combined reagent added to 50 mL sample, and the sample mixed?	4500-P E.4.a				
14) Are samples read at 880 nm within 10 to 30 minutes of adding combined reagent, using reagent blank as the reference solution? <i>Alternatively, 650 to 660 nm may be used, per flexibility in 4500-P F.2.a.</i>	4500-P E.4.a				
15) For highly colored or turbid waters, is a blank prepared by adding all reagents except ascorbic acid and potassium antimonyl tartrate to the sample, and is this blank absorbance subtracted from each sample absorbance?	4500-P E.4.b				
16) Are initial calibration curves composed of at least six standards?	4500-P E.4.c				
17) If daily initial calibrations are not performed, are daily continuous calibration verifications performed?	4500-P E.4.c				
Notes/Comments:					